

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Yasushi NOGUCHI et al.

Group Art Unit: 1791

Application No.: 10/584,943

Examiner: E. ROYSTON

Filed: July 5, 2006

Docket No.: 128634

For: METHOD FOR MANUFACTURING CERAMIC STRUCTURE

DECLARATION UNDER 37 C.F.R. §1.132

I, Yasushi NOGUCHI, a citizen of Japan, hereby declare and state:

1. I have a degree in Industrial Chemical Engineering which was conferred upon me by Okayama University in Okayama prefecture in 1988.
2. I have been employed by NGK Insulators, Ltd. since 1988 and I have had a total of 22 yeas of work and research experience in Ceramics materials.
3. I am a named inventor in the above-captioned patent application.
4. I have a professional relationship with the Assignee(s) of the above-identified patent application. In the course of that professional relationship, I received compensation directly from the assignee(s) for my work relating to ceramics development. I am not being compensated for my work in connection with this Declaration.
5. The foamed resin storage conditions of examples and comparative examples, the porosity of the resultant honeycomb filter, and an included gas amount were measured on the following conditions and by the following methods. I and/or those under my direct supervision and control have conducted the following tests to show the effects of storing a foamed resin on porosity in a ceramic structure.

- (1) A foamed resin was stored in a sealed constant-temperature tank set at 40°C.
- (2) An average pore diameter was measured with a mercury intrusion type porosimeter manufactured by Micromeritics Co., and a porosity was converted from a total pore volume (at this time, the true specific gravity of cordierite was set to 2.52).
- (3) The foamed resin was sufficiently dried in a desiccator, and a dried weight w_1 was measured. Thereafter, acetone which was a foamed resin solvent was added to the foamed resin, and the resin was dissolved to fly and scatter an included gas. After acetone and the included gas were sufficiently flied and scattered, a weight w_2 was measured. An included gas amount w_g (weight%) was calculated by the following equation.

$$w_g = (w_1 - w_2) / w_1.$$

To a cordierite forming material made of talc, kaolin, alumina, aluminum hydroxide and silica, there were added: 2.0 wt% of a copolymer foamed resin having an average diameter of 50 μm and a shell wall thickness of 0.2 μm , containing 60 wt% of acrylonitrile (AN) and 40 wt% of methyl methacrylate (MMA) (an included gas was a mixed gas of isobutane ($C_n = 4$) and isopentane ($C_n = 5$), a C5 component content was 95 wt%) and stored at 40°C for 4 weeks; 5 wt% of a water-soluble cellulose derivative; 0.5 wt% of a surfactant; and water. The material was kneaded with a kneader, and columnar plastic clay deaerated with a clay kneader was obtained. This columnar plastic clay was extruded to obtain a honeycomb body having a diameter of 300 mm, a partition wall thickness of 300 μm and a cell number of 300 in^{-2} . A dried body was cut into a length of 350 mm, and opposite end face portions were alternately closed with paste of the cordierite forming material to obtain a zigzag pattern. This body was fired in a single kiln firing furnace at a maximum temperature of 1420°C for 150 hours on schedule. As a result, there was obtained a

satisfactory honeycomb filter constituted of a cordierite fired body in which any crack was not generated during the firing.

The testing parameters and results of Example 1 are shown in Table 1 along with Examples 2-8 and Comparative Examples 1-5. In Examples 2-8 and Comparative Examples 1-5, cylindrical clay was prepared in the same manner as in Example 1 except that a resin constituting ratio of an outer shell of a foamed resin, an included gas amount, a C5 component content of the included gas and a storage period were changed as shown in Table 1. Table 1 shows a foamed resin storage period, the amount of the included gas before and after the storage, and the porosity of the resultant honeycomb filter. This clay was extruded into a honeycomb shape and fired to obtain a honeycomb filter in the same manner as in Example 1. Table 1 shows the amount of the included gas before and after the storage, and the porosity of the resultant honeycomb filter.

6. To achieve the porosity results of the Examples in Table 1, the thresholds must meet the critical requirements in the columns of Table 2 titled Foamed resin AN amount (wt%), Foamed resin MMA amount (wt%), Gas weight decrease ratio (%) and Included gas amount after storage (wt%).

Example 8 shows measurement evaluation for a bottom limit for an included gas amount after storage.

Comparative Example 4 is not acceptable because a gas weight decrease ratio is large due to a non-allowable threshold in an outer shell of a foamed resin, even though Comparative Example 4 satisfies a content of C5 component in included gas. Comparative Example 5 is not acceptable because an included gas amount before storage is substandard, even though Comparative Example 5 shows a threshold level in an outer shell of a foamed resin.

7 As a side note. Example 5 of Table 1 does not correspond to Example 5 in the Table in Applicants' specification. The reason the Example 5 in Applicants' specification is not included here is because the storage amount is only 1 week.

Table I

	Foamed resin characteristics							Amount of foamed resin to be added (wt%)	Content of C5 component in included gas (wt%)	Honeycomb porosity (%)
	Foamed resin AN amount (wt%)	Foamed resin MMA amount (wt%)	Included gas amount before storage (wt%)	Foamed resin storage temperature (°C)	Foamed resin storage period (weeks)	Included gas amount after storage (wt%)	Gas weight decrease ratio (%)			
Ex.1	60	40	12	40	4	9	25	1.9	95	65
Ex.2	80	20	12	40	4	10	17	1.9	90	65
Ex.3	90	10	9	40	4	8	11	1.9	90	67
Ex.4	90	10	13	40	4	11	15	1.9	95	68
Ex.5	62	38	15.9	40	4	13.5	15	1.9	85	69
Ex.6	60	40	13.8	40	4	10.0	28	1.9	95	66
Ex.7	60	40	16.1	40	4	13.1	19	1.9	95	67
Ex.8	60	40	11.4	40	4	8.3	27	1.9	95	65
Comp.Ex.1	50	50	11	40	4	4	64	1.9	60	63
Comp.Ex.2	50	50	11	40	4	4	64	2.4	60	65
Comp.Ex.3	55	65	12.3	40	4	7.7	37	1.9	75	64
Comp.Ex.4	58	42	9.3	40	4	6.4	31	1.9	85	63
Comp.Ex.5	60	40	4.4	40	4	3.2	27	1.9	85	62

Table 2

	Foamed resin AN amount (wt%)	Foamed resin MMA amount (wt%)	Included gas amount before storage (wt%)	Foamed resin storage temperature (°C)	Foamed resin storage period (weeks)	Included gas amount after storage (wt%)	Gas weight decrease ratio (%)	Content of C5 component in included gas (wt%)	Amount of foamed resin to be added (wt%)	Honeycomb porosity (%)
Desired	≥ 60 wt%	≤ 40 %			4 weeks	≥ 8 wt%	≤ 30 %	≥ 80 wt%	1.9 wt%	≥ 65 %

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date:

Mar. 5, 2010

Yasushi Noguchi
Yasushi NOGUCHI